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In-Situ Cure Monitoring of Composites by Fiber-Optic Molecular Sensors

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19 ABSTRACT (Continue on reverse if necessary and identify by block number)

In-Situ Cure Monitoring of Composites by Fiber-Optic Molecular Sensors. N. H. Sung, W. Dang, Tufts University, C.S.P. Sung, University of Connecticut. Recently, a new technique has been developed for monitoring cure reactions in network forming polymers by using reactive molecular sensors. Diaminoazobenzene (DAA), for example, generates sensitive signals in UV-Visible and fluorescence spectra as a function of cure reaction in epoxy, such as DGEBA/DDS system.

This describes our current efforts on the application of molecular sensors to composite cure monitoring via remote sensing fiber-optic probes. A custom-built fiber-optic fluorimeter allows on-line sensing of fluorescence signals directly from the cure environment. Different optrode configurations are developed to probe the structure at the local sites of surface, bulk, and interface. Single-filament distal-end probes are (see over)

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used for surface and bulk, while evanescent probes are utilized for the probe of interface regions.

Preliminary data of fluorescence are correlated with processing parameters, i.e. cure temperatures, and time, and molecular structure of epoxy network. The use of internal reference dyes for normalization of fluorescence signal is also discussed.

IN-SITU CURE MONITORING OF COMPOSITES BY FIBER OPTIC MOLECULAR SENSORS

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Recently, an approach of reactive labeling techniques was introduced for the study of polymerization and crosslinking in polymers (1-4). In this approach, the reactive label is designed to have similar reactivities as one of the polymerizing components, and to exhibit spectral changes in the region of UV-Visible absorption or emission. Basically, the substituent changes in the para and para' positions of conjugated aromatic labels as the reaction proceeds are the causes for such spectral changes. This approach has been applied to several polymers such as crosslinked epoxies (1), polyimides (2), polyurethanes (3) and polyamides (4). In the characterization of epoxy cure, a reactive label, p, p'diaminoazobenzene (DAA) showed significant red shifts in UV-Vis spectra and drastic enhancement of fluorescence intensity when reacted with epoxide.

In this study, we report on our attempt to apply DAA label for epoxy composites for in-situ cure monitoring using fiber-optic fluorescence instrument. In-situ cure monitoring is important during the manufacturing process at elevated temperatures and pressures since the properties of the composites depend strongly on the extent of cure. For the capabilities of remote sensing and optimization of cure, fiber optic sensors are desirable due to the small size, low weight and environmental ruggedness to enable their being embedded directly into composites. One example of fiber optic sensor is the work of Levy and Schwab who used viscosity dependent fluorescence intensity change for cure monitoring of epoxy based composites (5).

Fiber optic fluorimeter(Optical Sensor Consultants,Inc.),shown in Fig.1, was used to monitor fluorescence emission spectra as a function of cure time and temperature. Optical fibers with a diameter of 230 μm from Steve Brown Engineering were used. The same optical fiber was used for transportation of excitation and emission light. Optical probe configurations are varied for monitoring different sites, which are illustrated in Fig. 2. The epoxy system studied was a stoichiometric mixture of Epon 825 with diamino diphenyl sulfone (DDS) with a small amount of DAA (0.01% wt.). The tip of the fiber was immersed in the neat epoxy resin in a beaker which was placed in a temperature controlled oven.

Fig.3 shows the fluorescence emission spectra using fiber-optic fluorimeter for Epon 825-DDS-DAA system, when cured at 140°C. The fluorescence intensity due to DAA when it becomes a cross-link or branch point (1). Fig.4 shows the fluorescence intensity changes at emission maximum as a function of cure time at 140, 160 and 180°C. The higher the cure temperature, the faster the leveling off of fluorescence due to vitrification, as observed before (1). It is also seen in Fig. 2 that the fluorescence intensity decreases with increasing cure temperature, partly due to the temperature effect of

fluorescence of reacted DAA species. External factors such as excitation lamp fluctuations, optical alignment and the position of the filters were also found to influence the intensity.

In order to quantitatively correlate the fluorescence intensity to the extent of cure, we are in the process of establishing the temperature dependence of flourescence intensity as well as testing some highly fluorescent compounds such as 9,10-diphenyl-anthracene and perylene as in internal standard.

Acknowledgement

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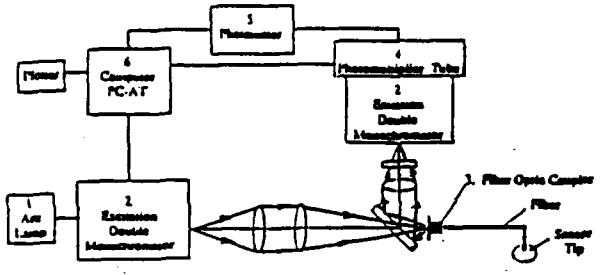

1. Quartz-Sapphire Xenon Lamp
2. Czerny-Turner Style (Double holographic grating)
3. Spindler and Hoyer Custom Design
4. Detector (185nm – 900nm)
5. Photon Counter consisting of pre-amp, discriminator, digital ratemeter and computer interface
6. Data Aquisition - IBM/AT, Color ... Software Supports five spectral types

Fig.1 Schematic Diagram of Fiber Optic Fluorimeter

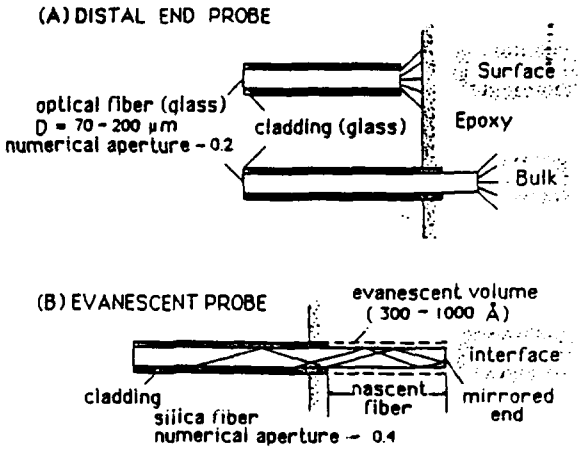

Fig.2 Probe Configurations for surface,bulk and Interface

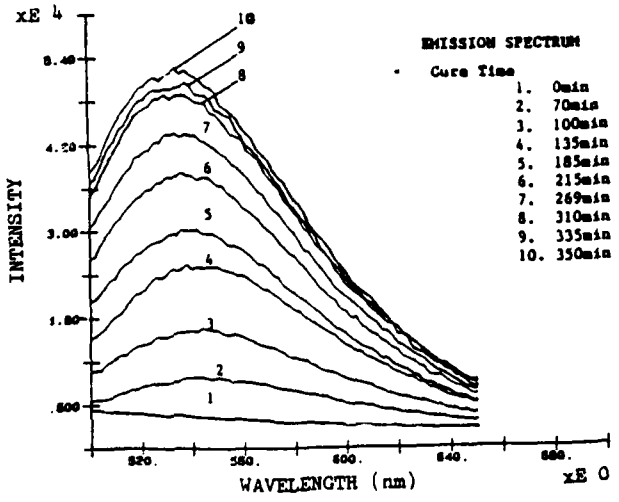

Fig.3 Fluorescence Emission Spectra for Epon 825/DDS/DAA During Curing at 140°C

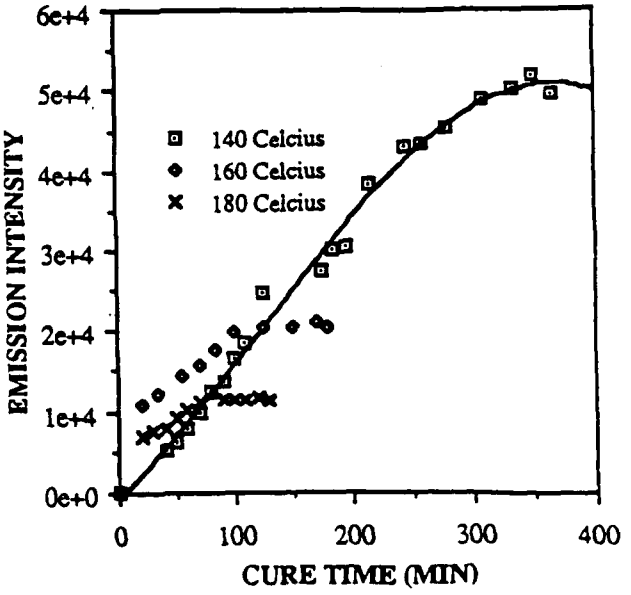

Fig.4 Fluorescence Emission Maxima vs. Cure Time at Three Different Temperatures